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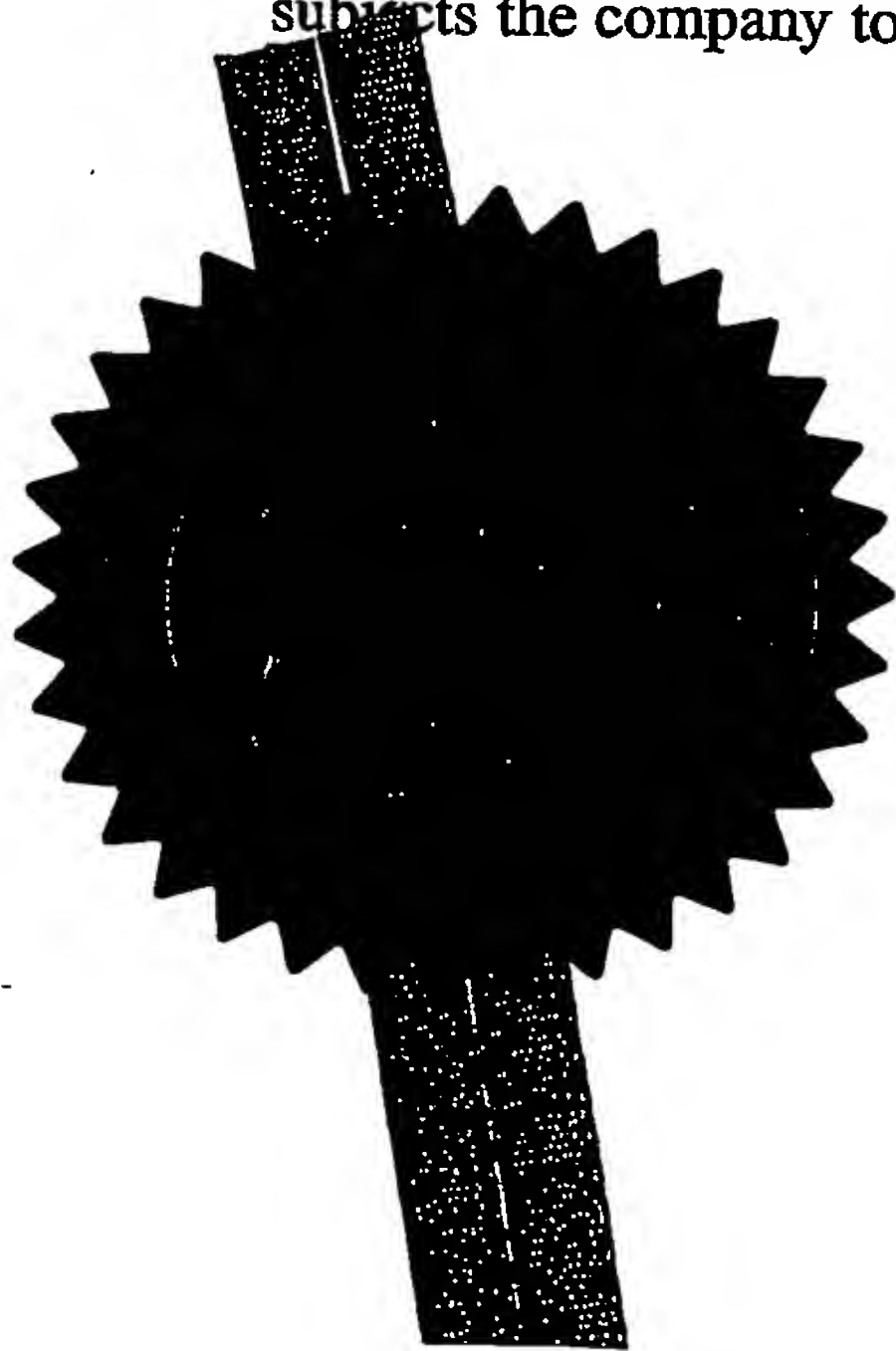
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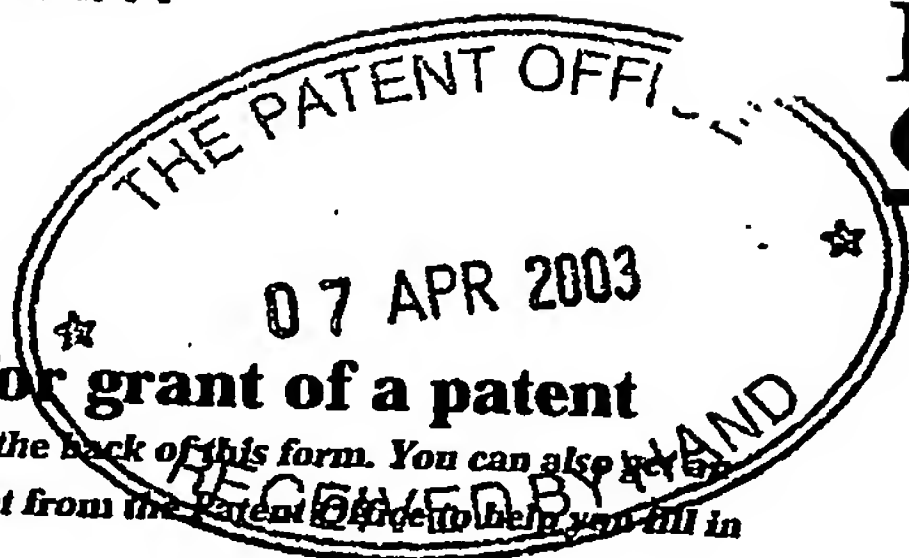


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08APR03 E/98450-S 002890  
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Cardiff Road  
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1. Your reference

RSJ07840GB

2. Patent application number

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0308021.5

3. Full name, address and postcode of the or of each applicant (underline all surnames)

Aerstream Technology Limited  
The Hart Partnership  
24 High Street  
Saffron Walden  
Essex, CB10 1AX  
United Kingdom

Patents ADP number (if you know it)

If the applicant is a corporate body, give the country/state of its incorporation

8605735001

4. Title of the invention

Spray Electrode

5. Name of your agent (if you have one)

Gill Jennings & Every

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

Broadgate House  
7 Eldon Street  
London  
EC2M 7LH

Patents ADP number (if you know it)

745002

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number

Country

Priority application number  
(if you know it)

Date of filing  
(day / month / year)

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application

Date of filing  
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8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:


YES

- a) any applicant named in part 3 is not an inventor, or
  - b) there is an inventor who is not named as an applicant, or
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Request for preliminary examination and search (*Patents Form 9/77*)

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(*Patents Form 10/77*)

Any other documents  
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NO

11. For the applicant  
Gill Jennings & Every

I/We request the grant of a patent on the basis of this application.

Signature



Date

7 April 2003

12. Name and daytime telephone number of person to contact in the United Kingdom

R.E. Skone James

020 7377 1377

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## **Spray Electrode**

**This is the description of a spray electrode for the atomisation and vaporization of chemicals through the generation of a high specific surface of a liquid in a device using electronic means. Patent application PCT/GB02/02900 describes one such device that can be used to generate atomised droplets of a liquid that are electrically discharged, and which do not deposit on the device itself. This is an extremely efficient system.**

**We have discovered through the course of experiment using this device and others like it, that certain chemicals react with spray electrodes during extended use, and in some cases this can lead to spray electrode degradation and spray electrode blockage. This can be particularly impracticable if the device is intended for long periods of use without servicing or observation.**

**Such problems are generic to all electrostatic spray devices where the spray electrode or spray electrode tip is situated during use in a strong electric field. Examples of other devices that would suffer in such circumstances include those described in patents WO92/15339, US5337963 and GB19780014967. For devices such as these, and others like them or based on them, the electric field can catalyse or be the direct cause of chemical degradation, either in conjunction with the spray electrode or independently.**

**Practical observation has indicated that some organic molecules, including some that are used in fragrances, have reacted to form solid residues at the tip of the spray electrode, and in some cases this has contributed to a gradual reduction in the performance of the device over time. Even the degradation of molecules to a lesser degree is undesirable, since for example a fragrance's character may change or an active molecule used for health reasons could become less potent or be rendered obsolete. It is possible to simply leave out from spray formulations those problematic compounds or delicate molecules, but this is limiting.**

Since the use of electric fields to generate droplets is desirable due to the performance and efficiency of such devices, it is important to reduce any deleterious effects of the electric field, such as chemical reactions, and to moderate or eliminate the consequences of such effects. Practical observation has shown that these chemical reactions are exacerbated by the presence of corona, so that attenuating the breakdown of air around the spray electrode limits these reactions and their effects. Therefore, to control corona, or at least to attempt to limit its occurrence to predetermined areas of a device it is essential to have a robust spray system with consistent performance.

In this respect it is important to dictate the electric field accurately - including the field created by the space charge of droplets and any ions. Therefore, it is also important to be able to predict the direction and spread of a liquid emitted from the spray electrode, and this must be consistent from one liquid to another. Some liquids form what are known as multiple jets, where more than one jet of liquid is formed at the spray electrode as described by Cloupeau and Prunet-Foch; *J Electrostatics*; 22 (1989) 135-159. The formation of these jets is difficult enough to predict for a single liquid, but over a range of different liquids with different properties there is little consistency at all. This means that a device would have to be set up specifically for each and every formulation, which is impractical for many applications.

The device described in this patent shows how by careful construction of the spray electrode and selection of its orientation with respect to the remainder of the device as well as its material composition, these problems may be overcome or reduced to acceptable levels. There are therefore provided a number of practical steps that pertain to both the causes and effects, which alone, or better still in combination, provide a more robust device that is less sensitive to formulation changes and whose performance is more consistent over long periods of use. These inventive features are described in the following paragraphs and practical examples are provided with reference to the figures.

According to one aspect of the invention there is provided a spray electrode and another reference electrode in close proximity, with an electric potential set up between



them, and where the spray electrode comprises a modified capillary structure such that the end exposed to the electric field includes a focal prominence to prevent or inhibit the formation of multiple jets.

Multiple jets are formed when a single jet can no longer sustain the whole flow rate and there is sufficient space and strength of electric field for additional jets to be formed. With the most common spray electrodes - formed from capillaries cut perpendicular to their axis - this means that the jet moves from a co-axial location where the field is strongest to the rim of the capillary where it is weaker. If this is still not sufficient to maintain the flow of liquid a second jet is formed at a diametrically opposite location on the rim of the capillary, (Jaworek & Krupa; *J. Aerosol Sci.* 7 (1996): 979-986).

The number of jets is very difficult to determine unless all aspects of the electric field within the device are known. This means that the electrical set-up, such as the geometry and voltage potential must be determined, but also the properties of the liquid and the flow rate of liquid to the nozzle, since these also effect the electric field. Such restrictions make the formation of multiple jets unhelpful in most commercial applications, and means to hinder or prevent the formation of multiple jets are extremely commercially advantageous.

This would seem to indicate that capillaries are therefore not the best form of spray electrode to use if multiple jets are not wanted. We have found, however, that with suitable modification they can be made to eliminate multiple jet formation.

Thus, a focal prominence should be added to the rim of the spray electrode. This feature focuses the electric field at one point on the spray electrode, so that the electric field there is sufficient to form a jet at the prominence, but is too weak elsewhere on the electrode for the formation of further jets. Furthermore, the prominence must focus the electric field sufficiently so that two or more jets cannot form at its location, and it is therefore important that its shape creates only a single point at which the electric field is a maximum - everywhere else the electric field should be much weaker.

Descriptions of suitable geometries are provided later, but one means is to create a local prominence at the tip of a capillary spray electrode by means of a small amount of

additional material in a rounded nipple, with a radius of curvature substantially less than that of the capillary itself. Although there are some examples of 'teeth' and other such features provided in the literature (US5503335, US5927618) these will not do at all since they actually create a plurality of localised electric field maxima. By contrast, those described in this patent have, by design, only one point of maximum electric field, and which are so shaped to inhibit the formation of multiple jets at the local prominence.

Another means to create a focal prominence according to the present invention is to create a bevel on the exposed end of a capillary. In this case the angle and finish of the bevel is important. For instance, forming a conventional bevel by grinding across the end will not do at all. This forms an edge like a hypodermic needle, which is far too sharp and acts not to focus the electric field to a point, but along the line of this 'knife-edge'. This is not effective for attenuating multiple-jet formation, since jets may easily form along the 'knife-edge'. Furthermore, a sharp edge created in this way increases the probability of excessive corona and this leads to inefficient use of the electrical power.

Instead it is necessary to round off the edge of a bevelled capillary spray electrode so that its radius of curvature is limited to a minimum of around  $5\mu\text{m}$ , and  $10\text{--}30\mu\text{m}$  being preferable.

We have found that chemical etching provides an ideal means of creating the required curvature and creates an even finish. The preferred chemical etchants used depend on the material of the spray electrode. Indeed, the material of the spray electrode itself is also important, since, for instance, coarse grain metals etch to a rough surface whereas more homogenous materials form smoother surfaces, which is advantageous.

According to a second aspect of this invention, we describe means of attenuating corona which also act as a source of uncertainty in the design of electrostatic spray devices. This may be achieved by modifying the surface of the spray electrode, for example, by creating a conformal coating over the spray electrode, having a low conductivity, and deposited using chemical, electrochemical or vapour deposition. Alternatively, the surface layers of the electrode itself can be modified to reduce its

conductivity. For example, with an aluminium spray electrode by increasing the oxide layer by anodising.

According to a third aspect of this invention the orientation of the focal prominence relative to the second electrode should be specified depending on the application and the design of the device. We have found that various orientations are useful in different circumstances, since any ions created at the spray electrode should aid the spray process.

Where there are discharging ions present the focal prominence is best placed either facing the discharge electrode or to one side or the other. When the prominence faces the discharge electrode any ions produced at the spray electrode help keep the system stable, by annihilating the discharging ions and in the case of a device as described in PCT/GB02/02900, these ions help keep the spray away from the device itself. However, placing the prominence at the strongest part of the electric field can sometimes also bring on excessive corona, so the spray electrode must be suitably treated to attenuate such effects as described above.

The consequence of ions with some electrodes is gradual degradation of the spray electrode itself. This can be exacerbated by the liquid being sprayed if it has a corrosive nature. Such corrosion generally results in a change of the shape of the spray electrode as the products of the corona attack it. In this respect the best position for the prominence is not facing the second electrode, since this side then is attacked first, and the changes in geometry that take place have a significant impact on the electric field and hence the spray behaviour. When the prominence is placed to one side or the other, perpendicular to the direction of the second electrode such effects are reduced, and this is often the preferred orientation.

The least favourable position in these situations is when the prominence is located in the far side of the secondary electrode, since the spray droplets move inside the path of the ions and must cross it to exit the device. This leads to unwanted instabilities.



If, however, the second electrode is not producing ions, but simply acting as a reference or counter electrode, the prominence is best placed on the far side, since the droplets are then encouraged to follow a longer path between the spray electrode and the counter electrode, which is usually preferable in all cases except when the spray is being used to coat the counter electrode, such as for paint!

Another aspect of this invention is to help reduce the build up of deposits or chemical reactions with the spray electrode itself using pressure waves inside the internal channel of the electrode. Although by inhibiting the formation of multiple jets one can increase flow rate and so better 'clear' the spray electrode, additional clearance may be achieved by applying a pulsed pressure wave to the liquid inside the reservoir. This wave reduces the probability of build up inside the electrode and is particularly useful with less viscous liquids where the viscous boundary layer at the inside surface of the electrode is small.

There now follows a description of the invention by way of example with reference to the accompanying drawings, in which:

Figures 1(a) and 1(b) illustrate an example configuration of electrodes embodying this invention and the elements required to create a working device.

Figures 2(a) to (f) illustrate schematically various different electrode treatments, giving examples of both good and bad practice.

Figures 3(a) to (c) illustrate schematically cross-sectional views of the tips of three electrodes, again providing examples of both good and bad practice.

Figures 4(a) to (c) illustrate schematically various orientations of the spray electrode as described in the present invention.

Figure 5 illustrates schematically a part cross-sectional view of one means for cleaning the spray electrode according to the present invention.

Figure 6 shows a cross-sectional view of one means of creating an integral spray electrode cleaning system.

Figure 1(a) illustrates schematically one possible embodiment of the present invention, where there is a spray electrode 1, and a reference electrode 2 that can also be a discharging electrode. The spray electrode 1 in this example comprises a 27-gauge, conductive capillary, such as an aluminium capillary, and the reference electrode is any conducting surface, such as a stainless steel sheet or pin.

Electrical connections 3 and 4 are made between the electrodes and a driving circuit 5, which delivers a high voltage with a constant or regulated current when activated. Liquid 6, held in a flexible reservoir 7 made from A PET film or laminate, (such as from a Mylar® laminate film manufactured by Dupont of Dupont Building, 1007 Market Street, Wilmington, DE 19898, USA), is simultaneously pumped along the spray electrode 1 by the pump 8. A suitable pump would be one such as described in patent US5961298, but any means of pumping liquid would be sufficient. The liquid is broken up into droplets by the electric field, which are sprayed charged or discharged depending on the nature of the reference electrode 2.

Figure 1(b) illustrates diagrammatically one possible electrical circuit embodying the invention for driving the device in Figure 1(a). A battery or other low-voltage power source 9 is connected via a control switch 10 to the input of a high-voltage converter 11. The output terminals of the converter are connected to the electrical conduits 3 and 4 of the device in Figure 1(a).

A simple converter for the device is a PSM10-103P manufactured by HiTek Power, Durban Road, Bognor Regis, West Sussex, PO22 9RL, UK. Note that any converter capable of delivering voltages from 1 to 30KV at roughly 10 $\mu$ A or less is suitable for this device. Higher power converters can also be used although they provide no added benefit and generally require more safety management. Low power devices, such as piezoelectric crystals or converters are ideal, and have distinct benefits such as reduced size and improved intrinsic safety.

It should be noted that there is no earth reference shown in the figure. This is deliberate since an earth reference can be omitted altogether or applied to any point in

the circuit unless the application requires one. The polarity of the high voltage output is also immaterial, although some rearrangement of the electrodes may be necessary, and for instance a PSM10-103P converter can be replaced by a PSM10-103N, (its negative counterpart), for a functioning device.

Figure 2 shows six variations of the tip of a spray electrode, where figure 2(a) illustrates a standard conductive capillary nozzle such as described in the art. In these examples the external diameter of the capillary is approximately 400 $\mu$ m and the internal diameter is approximately 200 $\mu$ m, i.e. the capillary is 27 gauge, although other gauges from 30 gauge and less are possible and the features would be scaled accordingly.

The nozzle in figure 2(a) produces multiple jets if the flow rate of liquid through the nozzle is high and the electric field around the tip is sufficiently strong. If the liquid cannot flow through a single jet sufficiently fast, second, third or multiple jets are formed at the tip:

Although under some circumstances this may be desirable, if control over the path of the droplets is required the unpredictable formation of multiple jets causes serious problems in such applications. Therefore means to hinder or prevent the formation of multiple jets are extremely commercially advantageous.

One means proposed here involves the creation of a focal prominence as a focussing point for the electric field, so that although a capillary may act as the final conduit of liquid to the tip of the spray electrode, the liquid does not spray off its end perimeter. The purpose of the focal prominence is to reduce the potential required to produce a spray by increasing the local electric field at a single point. This is achieved by for example the addition of a small nipple at the end of the capillary as illustrated in Figure 2(b), or by an additional rod, of smaller outer diameter than the capillary itself, fixed to the end of the capillary and substantially parallel to it, as illustrated in figure 2(c). In this later case the liquid travels over the end face of the capillary and onto the rod by surface tension forces of the liquid.

Note that the edges of the nozzles in figures 2(b) and (c) must have a slight radius and not be sharp, otherwise second, third or more jets may form at the end of the capillary diametrically opposite to the prominence or rod.

A focal prominence may also be obtained by cutting a capillary at an angle as illustrated in figure 2(d). However, this alone is not sufficient. A radius must also be applied to the edge as shown in figures 2(e) and (f), where the radius ranges from 5 to 50  $\mu\text{m}$ . Such radii are difficult to obtain by mechanical means, and if they are this leads inevitably to inconsistencies that provide additional local focal points in the electric field, which then form potential sites for multiple jets. It is therefore a feature of this invention that no such extra focal prominences are created - there should be only one at the point of maximum electric field.

It should be noted that the larger the radius applied to the end of the spray electrode the more acute the angle of bevel may be. When a minimum 5 $\mu\text{m}$  radius is applied to the tip of the spray electrode, for instance, the angle may be up to 60° or 70°. Whereas if it is 50 $\mu\text{m}$  the angle may be as low as 30° or 20°. Angles outside this range may be used, but their benefits appear greatest in between these values.

Figure 3(a) is a cross-sectional view through a capillary with a focal prominence provided by a bevel, again of roughly 27 gauge, although other gauges are possible but the features should be adjusted accordingly. Processing to this level is not sufficient to inhibit multiple jets and corona formation. Firstly, as the edge is so sharp, sometimes a nozzle like this will form two or more jets along the sharp, protruding edge. This is because in one direction - around the outer perimeter of the electrode tip - the radius of curvature is of the order of the capillary itself, but in a perpendicular direction it can be of the order of the atomic size. This means that a capillary like this acts as a sharp 'knife-edge', along which multiple jets may form.

Consequently, further modification of the electrode tip is required, to reduce the smallest radius of curvature, as illustrated in figure 3(b). Here the sharpest parts of the electrode tip have been etched away by means of suitable chemicals. For instance,

stainless steel electrodes may be etched by solutions of ferric chloride, hydrochloric acid, nitric acid or citric acid, for example, or a combination of these. Brass or copper based capillaries may be etched by ferric chloride solution or chromic-sulphuric acid mixtures. Aluminium capillaries may be etched by warm sodium hydroxide solutions or muriatic acid, for example.

Note that the concentration of the etchants and the etching time will depend on the material finish, such as surface roughness or the presence of machining lubricants, as well as its composition, granular structure and temper.

We have found a great deal of variation even for materials with apparently identical specifications, so it is recommended that tests be carried out on batch samples to determine the concentrations and etch times in every case. However, for the purposes of illustration, a 27gauge capillary of 304 stainless steel may be etched by 50% volume solution of concentrated nitric acid in water over 5 minutes. It is helpful to keep the etching time to the order of 5-10 minutes, so that the time required to first add the pieces and then rinse them after treatment does not become critical. Longer times are unnecessary and may be reduced by using a more concentrated solution, whilst conversely fast etch times may be made longer by reducing the concentration of the etchant.

It should be noted that by eliminating sharp edges in this way, excessive corona may also be prevented. Although corona are not always a problem, they are a source of electrical inefficiency. Only a very small number of ions are required to discharge the droplets – usually less than  $1\mu\text{A}$ . So corona with currents much higher than this only increase the power required by the spray unit, but do not enhance its functionality.

Figure 3(b) illustrates a modified nozzle that has further been coated in a thin layer of dielectric or semi-conductor. Such treatment inhibits the local formation of corona and can keep the power consumption down.

Once a nozzle has been suitably modified in the ways described above it then acts as a source for a single spray. Modifications that focus the electric field on one side or the



other mean that the orientation of the nozzle with respect to a second electrode become significant.

Figure 4(a) illustrates how by placing a focal prominence on the far side of a second electrode the path of charged droplets may be increased. If the focal prominence were placed on the near side the spray would travel more directly from one side to the other. This has benefits, for instance, in the charged delivery of aromas, where the longer the charged droplets are in the air the more they have evaporated before they inevitably land on the second electrode.

Discharging the spray stops the droplets from travelling to the second electrode, and so in this case a lengthening of the spray path is less necessary. In this case, by placing the focal prominence on the near side where the electric field is stronger, (being nearer the second electrode), its multiple jet attenuating properties are enhanced.

Where there is a chance that the latter orientation might induce excessive corona, the focal prominence may be placed at right angles, as illustrated in figure 4(c), or at some angle between the extremes illustrated by figures 4(a) and (b).

We have found that the break-down of the air around nozzles of this kind is often inevitable, although the degree of ionisation depends greatly on the shape of the spray electrode, the presence of a discharging electrode, and the flow rate of liquid. For example, lower flow rates ( $\sim 1\mu\text{l/s}$  or less); and the presence of any discharging electrode each raise the probability of gas discharge or corona.

The effect of this ionisation or the products of the ionisation, lead to chemical reactions at the tip of the spray electrode. This is not really an issue for the liquid being sprayed, since it is replaced regularly, and so any degradation is insignificant and often undetectable. However, the spray electrode itself is not replaced, and it can undergo gradual change over long periods of use.

We have found that there are various ways to overcome this degradation or to remove the products of such degradation before they impair the proper functioning of the electrode. One is to use a material for the spray electrode that is not sensitive to such

attack. Platinum or tungsten electrodes may be used for example or even aluminium. These are relatively expensive options compared with stainless steel, which is produced in a suitable capillary form for many medical and other uses.

Another means to counter the gradual build up of the products of nozzle and liquid degradation is to include in the liquid to be sprayed a chemical that slowly cleans the nozzle. One such example is citric acid which may be used to slowly clean a brass or stainless steel nozzle as it is used, or silicone oil to protect the surface.

Unfortunately, it is not always practical to use such chemicals since other compounds may be toxic in large doses by inhalation, for example. This patent therefore describes a method of integral ultrasonic cleaning that may be used to dislodge any products of degradation. Figure 5 shows how a cleaner unit 51 may be placed in-line between the pump 5 and spray electrode 1. The cleaner should comprise a means to add high a frequency pulse to the flow over the general flow to the spray electrode.

One means is to use a piezoelectric diaphragm 61 in series with the pump and electrode (not shown). The diaphragm needs to be pulsated with an alternating voltage 62 at a frequency of anything from 1kHz to 1MHz. These figures are just guide lines and frequencies outside this range may also be used. However, lower frequencies may create fluctuations in droplet diameter at the outlet, and higher frequencies may have negligible effect on liquids which are either extremely dense or viscous or both.

A suitable diaphragm is the 7BB-12-9 manufactured by MURATA ELECTRONICS (UK) LTD, Oak House, Ancells Road, Ancells Business Park, Fleet; Hampshire, GU51 2QW, United Kingdom. Since the build up of products due to degradation of the nozzle occurs over a relatively long period of time (days or weeks), it is only necessary to pulse the cleaner very occasionally. Usually once a day will be sufficient, and it is best to pulse it while the spray electrode is spraying. However, if the cleaning process effects the quality of the spray it may be pulsed just before spraying is initiated, so any dislodged products are removed before they have time to settle.

Note that it is possible to use this kind of action to also pump the liquid as described in patent US5630709. This pump has reciprocating parts that would impart

some high frequency pulses to the general flow of liquid, which could be used to clean the spray electrode while the device is spraying. In this case the pulsations can be smaller in amplitude since they are applied all the time the device is spraying. Such a set-up has practical and commercial benefits since it combines two actions into one component, thus reducing the number of parts, making the device cheaper to manufacture.

An alternative to piezoelectric components is to use a multi-headed peristaltic pump, (such as the REGLO Digital MS-2/12 manufactured by ISMATEC SA, Labortechnik - Analytik, Feldeggstrasse 6, 8152 Glattbrugg, Switzerland), as the main pump 5 in figure 5. In this case there are already pulsations superimposed on the main flow and this can be sufficient to clean the spray electrode. In this case the cleaner 51 and pump 5 are combined in a single component. However, large pulses may effect the quality of the spray, so this is better employed where precise control of the diameter of the droplets is not essential.

These steps for creating consistent performance from a spray device apply to all sorts of electrostatic devices, where strong electric fields exist around electrodes spraying a liquid. Examples include the dispersal of aromas, pest control agents or other compounds, such as for healthcare, pharmaceutical or other applications where inhalation is desired or required.

Examples of chemicals or mixtures thereof that can be used to clean the nozzle while it is spraying include liquid mixtures containing acids such as citric acid, nitric acid, muriatic acid, chromic acid, sulphuric acid, caprylic acid, cholic acid, decanesulfonic acid, deoxycholic acid, glycocholic acid, glycodeoxycholic acid, lauric acid, lauroylsarcosine, linoleic acid, linolenic acid, oleic acid, palmitic acid, palmitoleic acid, stearic acid, taurochenodeoxycholic acid, taurocholic acid, taurodehydrocholic acid, taurodeoxycholic acid, tauroolithocholic acid, tauroursodeoxycholic acid, and salts thereof; alkalis such as sodium hydroxide; detergents such as phospholipids, polyoxyethylene ethers such as the "Brij®" series produced by ICI, (ICI, 20 Manchester Square, London, W1U 3AN, UK),

ICI's "Synperonic®" series, ICI's "Tween®" series of non-ionic surfactants; and other compounds such as butylated hydroxyanisole, ferric chloride, ethanol, methanol, ether and isododecane; and compatible mixtures thereof.

Examples of compounds or products that could be used to protect the nozzle include surface active agents such as lauric acid, linoleic acid, linolenic acid, oleic acid, palmitic acid, palmitoleic acid, stearic acid; oils such as silicone oil, mineral oil; alcohols including methanol, ethanol; and compatible mixtures thereof.

Other applications and modifications thereof will be apparent to persons skilled in the art.

Figure 1(a)

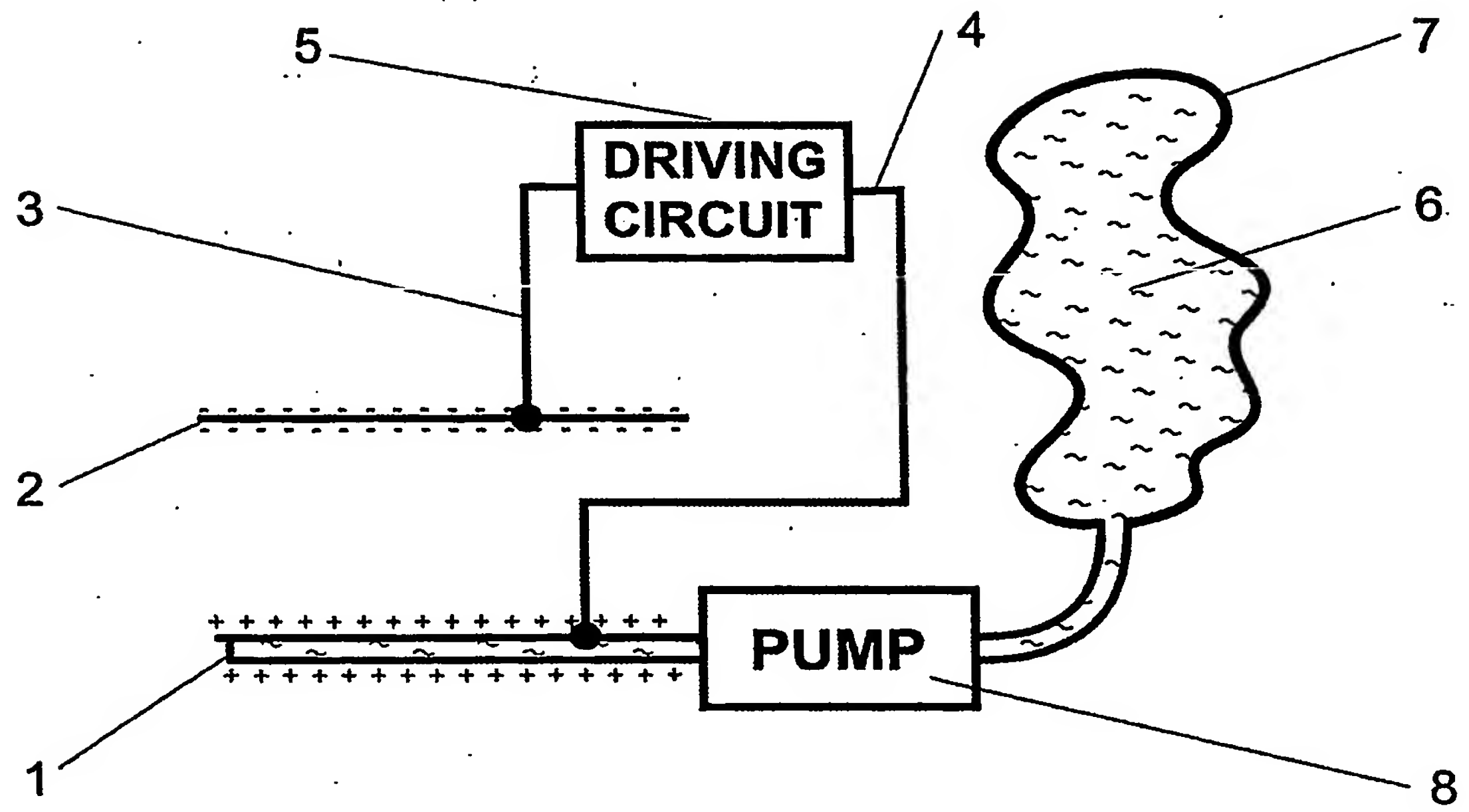


Figure 1(b)

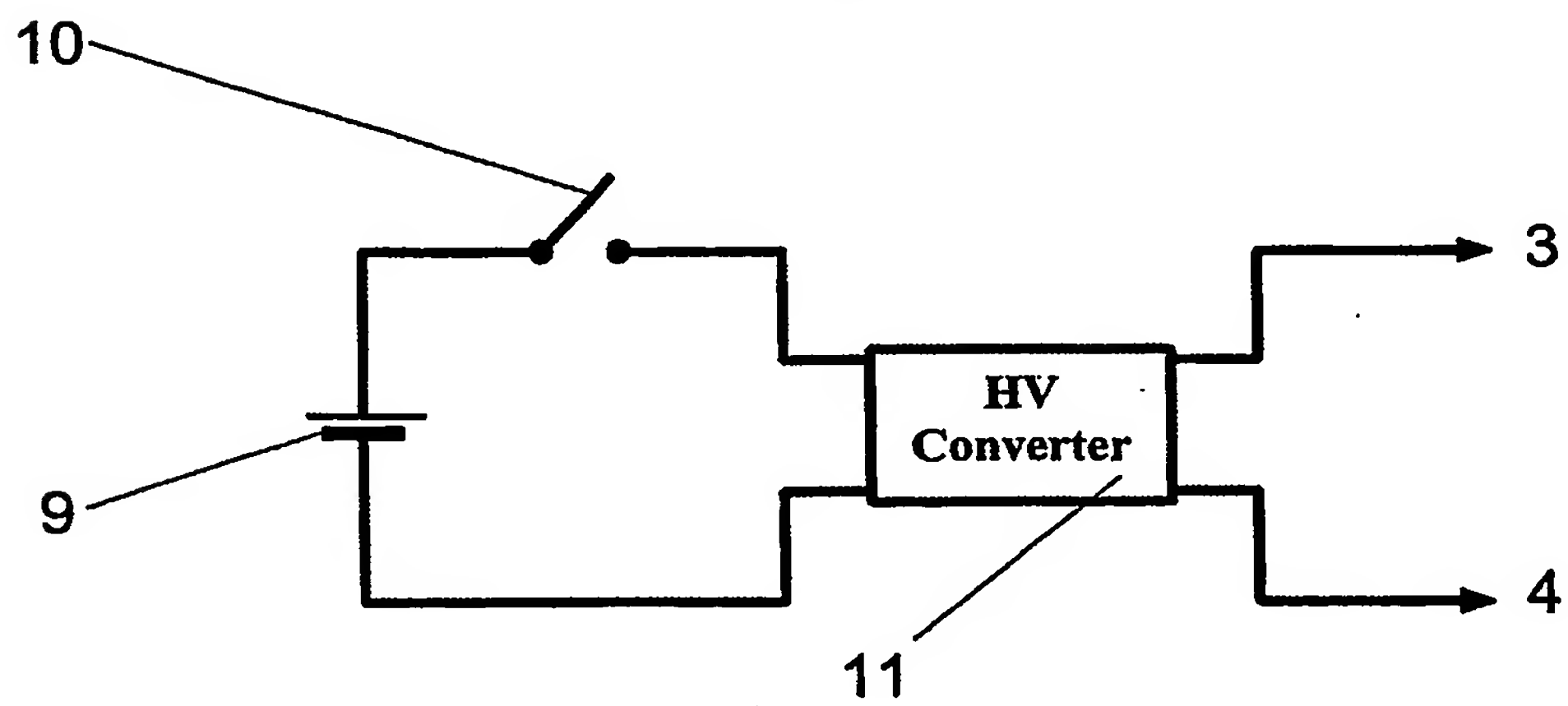




Figure 2

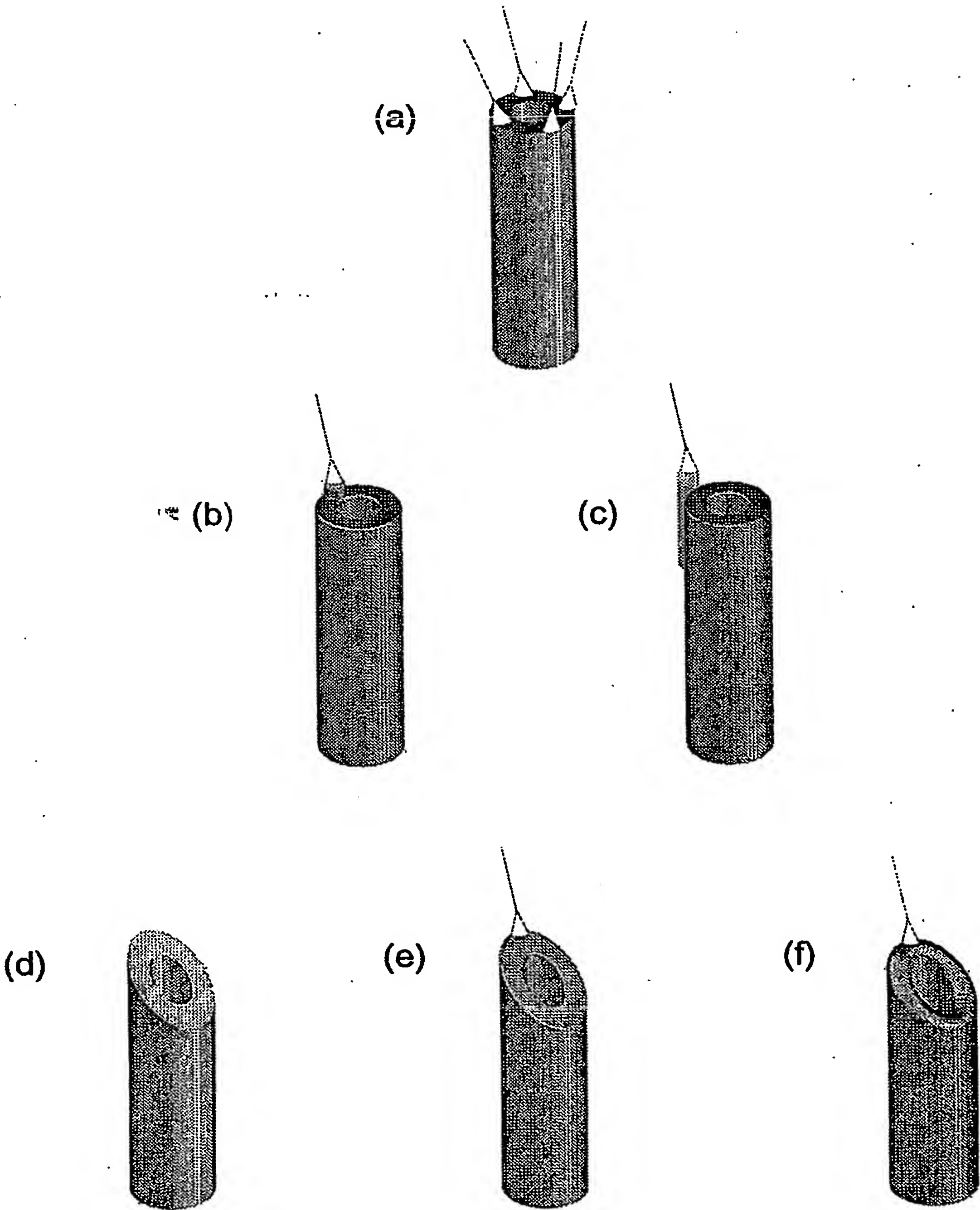
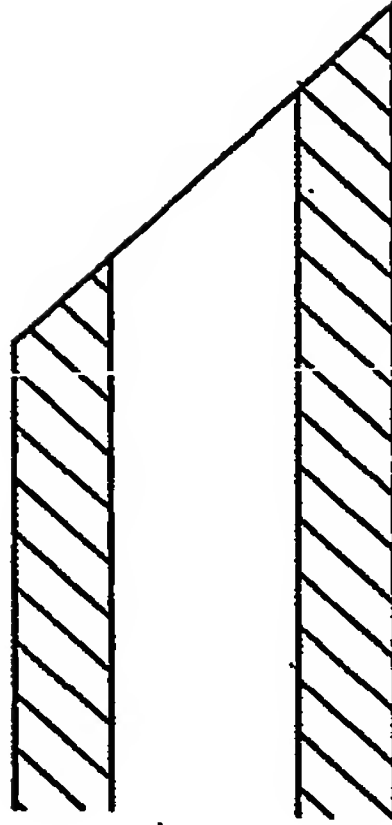
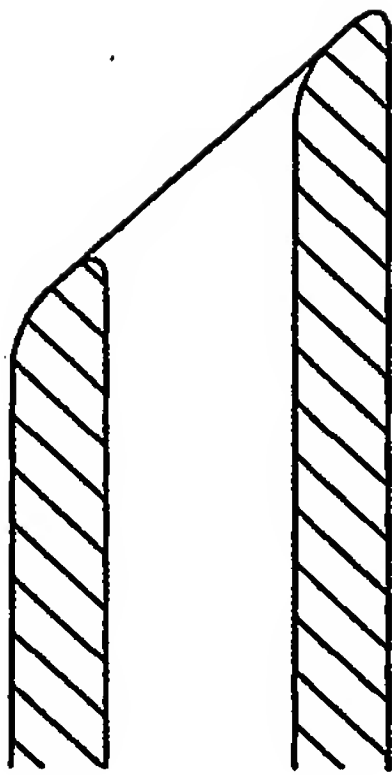


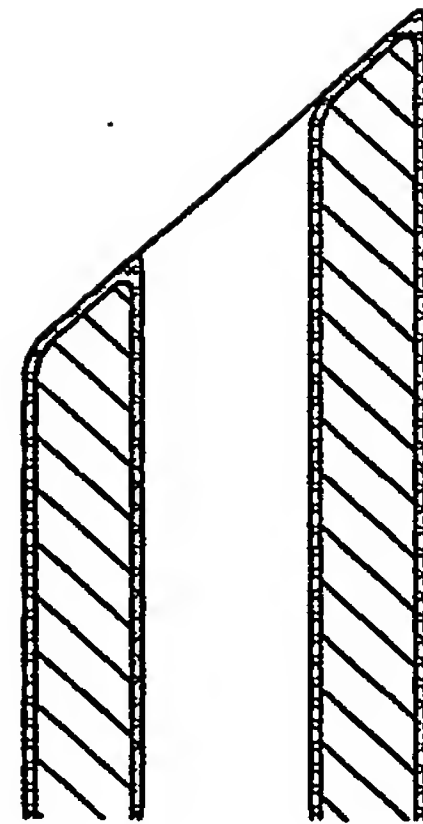
Figure 3



(a)



(b)



(c)

Figure 4

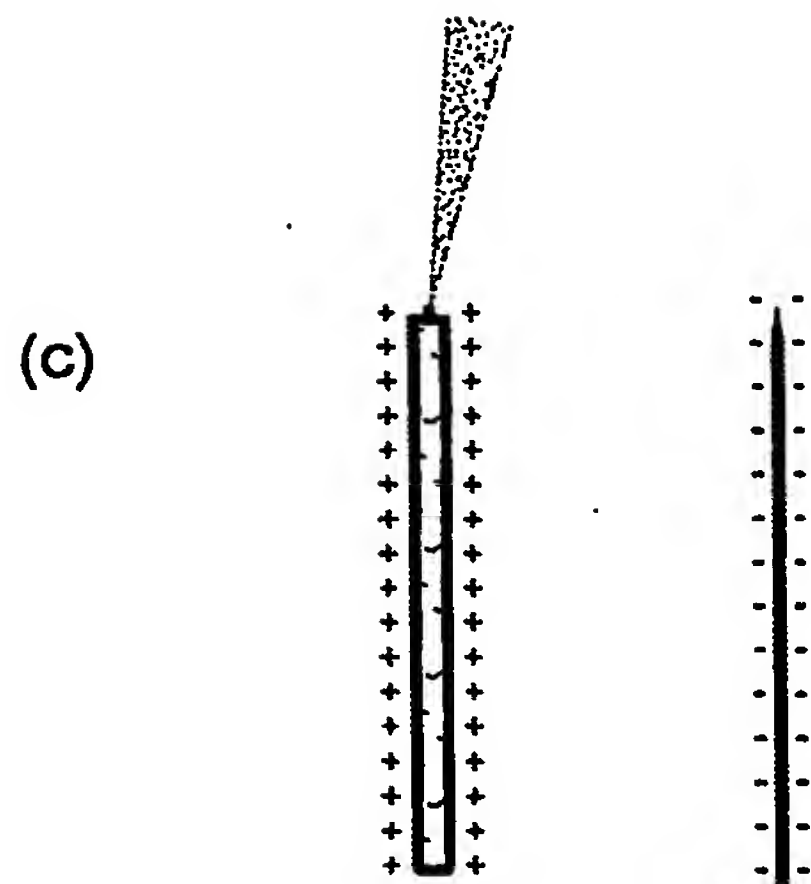
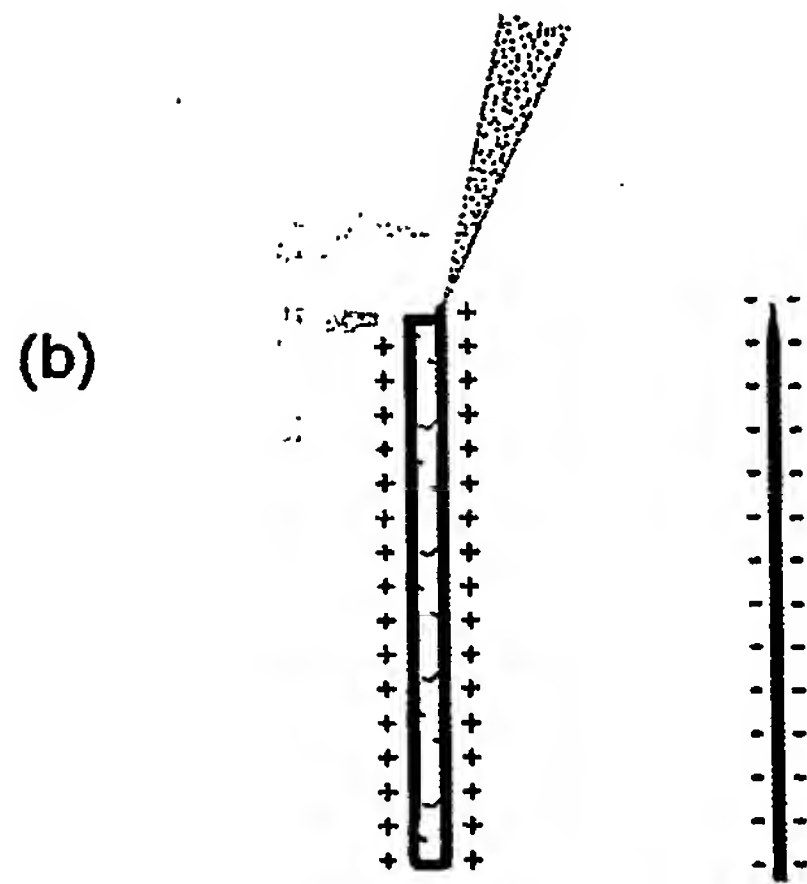
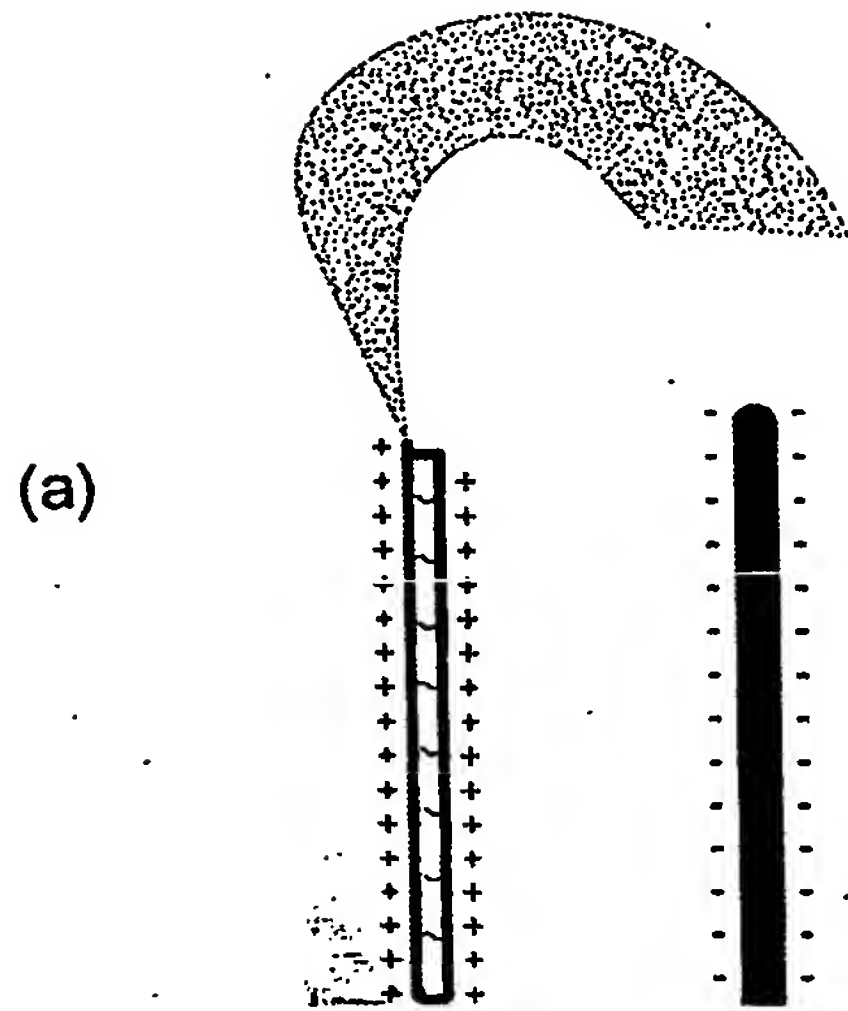


Figure 5

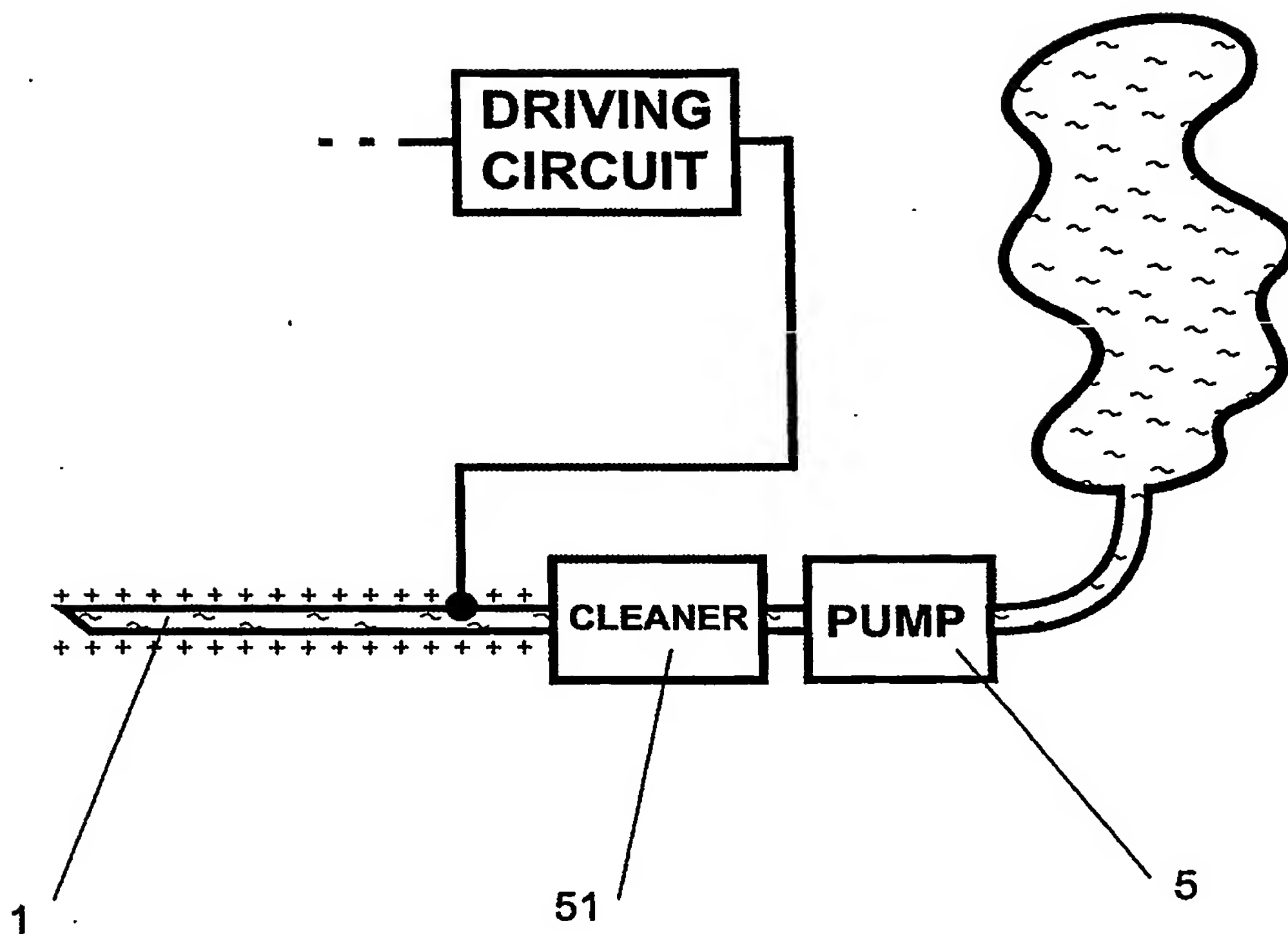
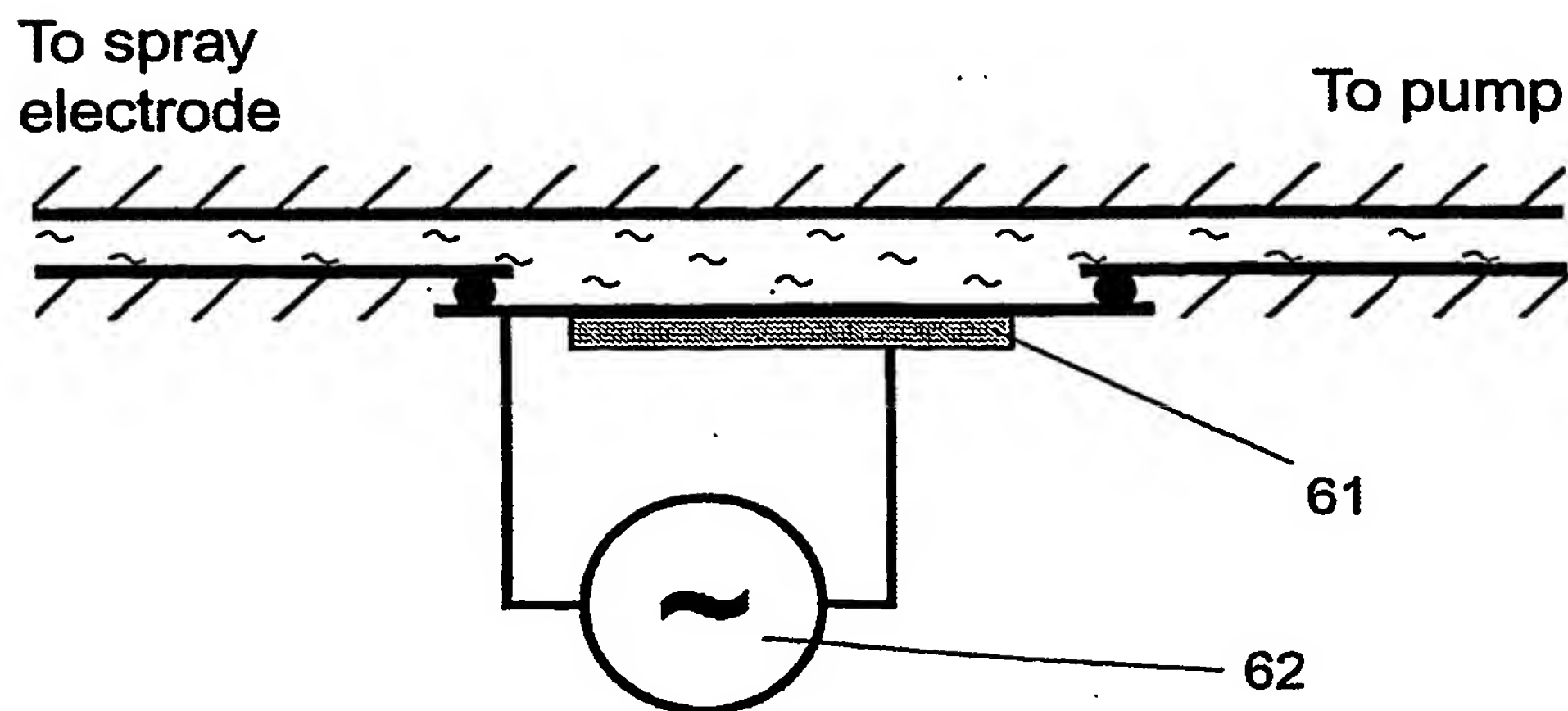
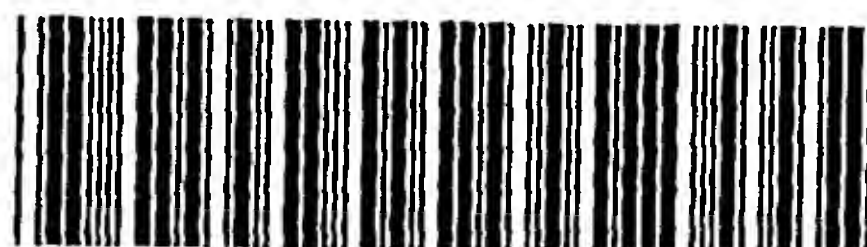


Figure 6



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